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Towards the origins of over-dispersion in beta source calibration

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ABSTRACT

The ability to deliver accurate and precise calibration doses is a central part of all trapped charge dating methods. Usually, the radiation source (α, β, X-ray) used to deliver these doses is, in turn, calibrated against an absolutely known reference source (usually a γ source) and many laboratories make use of Risø calibration quartz for this purpose. We have previously described this material in detail (Hansen et al., 2015) and discussed the over-dispersion (OD) of 3.2 ± 0.3% in calibrated dose rate observed over 16 years. This dispersion highlights the danger of relying on individual calibrations, and is clearly undesirable. Here we continue our investigation into providing reliable calibration materials for trapped electron dating. A comparison of the apparent quartz β source dose rates shows that there is no significant dependence on the geological source of the quartz. However the β dose rate decreases by 25% with increasing grain size from about 100 μm to 1 mm, and backscatter leads to a dose rate increase of ~1% per unit atomic number of the substrate. It is concluded that, for the multi-grain aliquots used in this study, the contributions to dose rate variability from grain size and substrate variations are likely to be negligible. Nevertheless there may be a practical advantage in using a high Z substrate because of the higher dose rate. Finally we test the measured to given dose (dose recovery) ratio for five heated feldspar samples and use the $pIRIR_{290}$ signal for β source calibration; surprisingly this gives an apparent β dose rate 15% lower than that to quartz despite their almost identical stopping power and mass absorption characteristics. Our results are discussed in terms of their significance for reproducibility and accuracy of β dose-rate estimates.

1. Introduction

Luminescence dating depends on an accurate and precise calibration of dose rate relevant to laboratory irradiations. In most routine analytical procedures for the measurement of natural dose, known laboratory doses are given using a radiation source (usually a β source) mounted on the luminescence reader. This local source must then be cross-calibrated against some independently calibrated standard, usually a γ source. In practice, the phosphor of interest is first given an accurately known γ dose by exposing it to a γ source of independently known dose rate (e.g. Bos et al., 2006; Hansen et al., 2015). The luminescence signal (TL or OSL) resulting from this known γ dose is then matched with that resulting from a timed irradiation using the β source to be calibrated, and the dose rate calculated by proportion, i.e. $D_\gamma \chi_\gamma = t\dot{D}_\beta \chi_\beta$. Thus

$$\dot{D}_\beta = \frac{D_\gamma \chi_\gamma}{t \chi_\beta} \approx \frac{D_\gamma}{t} \quad (1)$$

where D_γ is the dose absorbed from the γ source, and χ_γ and χ_β the luminescence sensitivity to γ and β radiation, respectively. $\dot{D}_\beta$ is the β source dose rate, and t the β irradiation time required to match the luminescence produced by the γ irradiation. Because β particles and γ rays are known to deposit energy through similar mechanisms, χ_γ is usually assumed to be indistinguishable from χ_β, and so the β source dose rate reduces to D_γ/t.

Irradiated quartz grains suitable for such a calibration procedure (calibration quartz, CQ, Hansen et al., 2015) have been produced in our laboratory for many years, and these have been widely used by the international community since before 1990. Kadereit and Kreutzer (2013) pointed out that the measured dose in our CQ may depend on the parameters chosen in the measurement procedure. Nevertheless, Bos et al. (2006) showed that, using a standard SAR protocol (225 °C preheat for 10s; 200 °C cut-heat; 125 °C stimulation with blue light) the β source calibration derived from our Batch 8 prepared in 2004 was indistinguishable from that derived from an independently prepared and γ irradiated quartz sample from their laboratory. However, Porat (p.c., 2015) has since reported an apparent 30% difference in calibration based on Batch 17, compared with that from both 57 and 71. Rhodes (p.c., 2015) reports a single-grain dose-rate difference of 12% between Batch 71 and 98, and Huot (p.c., 2012) up to 21% difference between Batch 17 and 60. Even within our own laboratory apparently systematic batch-to-batch discrepancies of up to 10% have been observed. The reliability of the β source calibration is a very serious issue for our community and so we have initiated investigations of the accuracy and precision of our calibrations, both past and present.

Hansen et al. (2015) presented the reproducibility of various

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batches of CQ over > 13 years and we have since extended this data set by three more years (Fig. S1a) and five additional new batches of calibration quartz. The average standard deviation from an exponential decay (Fig. S1, solid line) is 3%, and the ratio of the fitted decay constant to that of ^{90}Sr is 0.96 ± 0.05. The average dose rates have an over-dispersion of 2.8 ± 0.7% around the fitted line (Fig. S1b), and the maximum deviations of individual calibration measurements from the fitted line are as large as 10%.

Here we describe further investigations into laboratory parameters that might affect the accuracy and precision of our CQ. We first compare the apparent β source dose rates based on quartz derived from different geological sources and then investigate the dose rate dependence on grain size and atomic number of the sample holder used to hold the phosphor grains. After testing the dose recovery ratio for five heated feldspar samples, the $pIRIR_{290}$ signal is also used for β source calibration and the resulting apparent dose rate compared with that derived using quartz. This result is then compared with that predicted from Monte Carlo modelling. Finally our observations are discussed in terms of significance for reproducibility and accuracy of laboratory β dose rates.

2. Instrumentation

All luminescence measurements were undertaken using a Risø TL/OSL DA-20 luminescence reader fitted with blue LEDs (470 nm, ~80 mW/cm^2) and a ^{90}Sr/^{90}Y β source (Bøtter-Jensen et al., 2010). For measurement, a monolayer of quartz grains were mounted on 0.3 mm thick stainless steel discs using silicone oil. OSL signals were derived from the first 0.64 s of stimulations minus the subsequent 0.64 s. Equivalent dose measurements employed a SAR protocol (Murray and Wintle, 2000, 2003) with a 260 °C for 10 s/220 °C preheat/cutheat, stimulation at 125 °C and cleanout with stimulation at 280 °C for 40 s. XRF measurement made use of a Risø XRF attachment to the reader (Kook et al., 2012) specifically designed for characterising the composition of quartz and feldspar extract (e.g. Porat et al., 2015). All XRF feldspar grains were measured as loose grains on Mo cups.

3. Apparent dose rate dispersion using quartz of different origins

Calibration quartz has been produced in our laboratory for over 40 years and during that time, several different parent sediments have been employed. Although all these came from the west coast of Jutland, sediment in this region can derive from a variety of geological provinces. Accordingly the possible contribution to batch-to-batch variability from different sources of quartz was examined by investigating whether the apparent dose rate depends on the geographical origin of the quartz extracts. To increase the chance of differences in geological provenance, 9 different quartz samples were selected from various widely-separated locations around the world; these were then sensitised by heating to 700 °C for 1 h. The latter also ensures that the OSL signals are dominated by the fast component. They were then given a 2 kGy ^{60}Co gamma dose followed by heating to 450 °C for 1 h to empty the dosimetry trap (Hansen et al., 2015). The suitability of these samples for use as calibration quartz was then confirmed by irradiating aliquots of each sample with a β source for a known length of time, and then measuring this given dose in the usual manner. The results of this β dose recovery test are summarised in Fig. 1 (green symbols), and it can be seen that all dose recovery ratios lie well within ± 10% of unity; the mean ratio is 1.008 ± 0.013 (n = 9). Nevertheless two of the nine results (Namibia, France) are statistically inconsistent with unity. The nine sensitised samples were then placed individually in glass tubes (2.25 mm wall thickness) and irradiated at the same time 2.0 m from a calibrated point ^{137}Cs source in a scatter-free geometry (Hansen et al., 2015), to give an absorbed dose of 4.81 ± 0.07 Gy.

The apparent dose rates derived from these nine samples are summarised in Fig. 1 (black symbols). All individual calibrations are

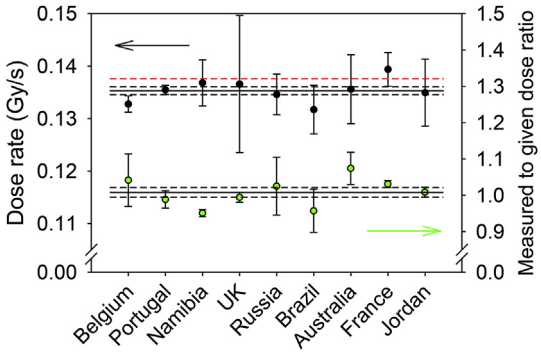

Fig. 1. Top: β source dose rate derived using nine different quartz samples from widely varying locations around the world. Red dashed line represents average beta source dose rate from Batch 90. Bottom: dose recovery ratio for the same samples. All data represents the average of six 8 mm aliquots measured per sample, and error bars represent one standard error. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

consistent with a mean dose rate of 0.1353 ± 0.0008 Gy s^{-1} (n = 9); this includes the two samples for which the dose recovery ratios were not consistent with unity. For reference, the mean dose rate based on Batch 90 (0.1376 ± 0.0012 Gy s^{-1}, n = 28) is shown in red (see also Fig. S1). It is concluded that there is no evidence for a dependence of apparent dose rate on the origin of the quartz extract.

4. Grain size and substrate dependence

4.1. Grain size

One possible cause of variation in apparent dose rate from aliquot to aliquot, and from sample to sample, is variation in mean grain size. The dose rate to grains of different sizes will be affected by Z-dependent backscatter (Autzen et al., 2017) and build up effects (Wintle and Aitken, 1977). In the preparation of standard calibration quartz, all grains are sieved to 180–250 μm, but that still allows a possible range in grain diameter of > 30% compared to the mean. Because of this, we have revisited and extended earlier investigations of the dependence of the apparent β source dose rate on grain size (e.g. Armitage and Bailey, 2005; Mauz and Lang, 2004). Various grain size fractions (see Fig. 2) were separated from the parent sand H33052 (Rømø, Denmark, used since Batch 90), except for the fractions in 300–1000 μm range, which were extracted from sample 178109 (aeolianite from Oitavos, Portugal). All fractions were pretreated and sensitised in the usual manner (section 3). After checking for the absence of a significant feldspar signal, dose recovery experiments were undertaken using each grain size (Fig. 2a). While most of the dose recovery ratios are within 10% of unity, there is a systematic trend of decreasing ratio with increasing grain size, and for the grain size intervals 500–800 μm and 800–1000 μm, the ratios fall below 0.9. These grain size fractions were then given an accurately known gamma dose as described above, and the apparent β dose rates derived. The unfilled circles in Fig. 2b show this observed grain-size dependence. The filled circles in Fig. 2b show these observed dose rates divided by the appropriate dose recovery ratio. This is appropriate here (in contrast to natural doses, for which the evidence is ambiguous) because the two experiments are directly comparable, except that in one experiment both doses are given by a β source, and in the other the first by a γ source. Thus it is very likely that any systematic difference observed in one experiment will also be present in the other. There is also a further point at 1.0 mm, based on 1 mm thick quartzite rock slices (dose recovery given in Fig. 2a), and after correction this lies on the smooth curve defined by the smaller grain sizes. The initially flat or slightly rising part of the curve was explained by Wintle and Aitken (1977) as the effect of build-up, where

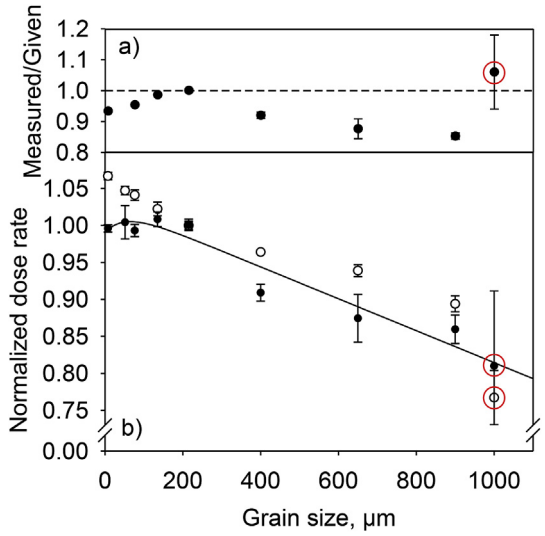

Fig. 2. a) Beta dose recovery as a function of grain size (4–11, 40–63, 63–90, 90–180, 180–250, 300–500, 500–800, 800–1000 μm); red circle – 1 mm thick rock slice drilled from quartzite (unknown origin). b) Dose rate dependence on grain size, for grains mounted in stainless steel discs; red circle - 1 mm rock slice. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

the electron flux (and so the dose rate) increases as the beam passes from air to quartz. The subsequent decrease with grain size is to be expected from the larger average contribution from low energy backscatter to smaller grain sizes (Autzen et al., 2017) and the greater attenuation of the primary spectrum as the grain size increases. Similar data have been reported by Armitage and Bailey (2005) and Mauz and Lang (2004). From this curve it can be predicted that the dose rate to 180 μm grains should be ~1.5% greater than that to 250 μm; this is small compared to the observed range of 10% in apparent β dose rate reported above.

4.2. Substrate

The substrate on which grains are irradiated contributes to the total beta dose rate by scattering incident electrons back into the quartz grains. When calibrating a β source, grains are usually presented either loose in a thin metal cup (usually 0.1 mm stainless steel) or adhered to a flat metal disc (0.3 mm stainless steel or aluminium) using silicone oil. The main advantage of the cup is the ability to hold a large sample (up to ~20 mg) but this is at the cost of significant dependence of the mean dose rate on sample mass, because the backscatter from quartz is different from that from stainless steel. Murray (1981) was the first to investigate this, and he showed that for an infinitely thick substrate (i.e. a substrate thickness very large compared to the range of the incident particles) the apparent β dose rate to CaF_2 (mean Z = 12.7) on aluminium (Z = 13) was ~13% lower than on steel (Z~26), and Greilich et al. (2008) and Autzen et al. (2017) have since modelled these data. Since the effective Z of quartz (Z~10) is slightly less than that of aluminium, the dose rate to a quartz grain sitting directly on steel will thus be considerably more than that to a grain sitting on other quartz grains. In practice, we do not irradiate on infinitely-thick substrates, and so we have investigated the variation in apparent β dose rates to be expected for a monolayer of quartz grains placed in 0.1 mm thick cups made from a variety of substrates (Fig. 3a). The variation in dose rate compared to that for grains irradiated on steel is considerable, from ~0.78 for aluminium, up to ~1.4 for platinum. These effects are considerably larger than those described earlier, presumably because in thin substrates density effects become important; for a given thickness of metal, there are 40% more atoms per unit area in a steel cup than in one made from aluminium. In addition, the β dose rate to 180–250 μm grains in platinum cups is ~40% higher than in conventional stainless steel cups. This increase offers the possibility of a greater routine beta dose rate in dating applications, but this can only be taking advantage of if the net spectrum to which the grains are exposed during irradiation is at least as suitable as that experienced on steel and aluminium.

Autzen et al. (2017) undertook Monte Carlo modelling (using GEANT4) of the incident β source spectrum and the backscattered spectra from infinitely thick substrates of various Z. These show that the main effect of changing Z is to change the intensity of the backscattered spectrum, but that there is a relatively small effect on the energy distribution. In Fig. 3b, we present the primary β spectrum and backscattered spectra using different substrates (all except Pt, Mo are from Autzen et al., 2017). For comparison, we have also calculated the infinite matrix β spectrum from ^{4} K, as representing a natural spectrum. It appears that a standard total laboratory spectrum (primary + backscattered) derived from irradiation on steel discs is likely to contain many more high-energy electrons than any infinite matrix beta spectrum. It is thus likely that the total spectrum resulting from irradiation on a high Z substrate (e.g. Pt, Z = 78, Fig. 3a) will be closer to the natural spectrum that those on lower Z substrates.

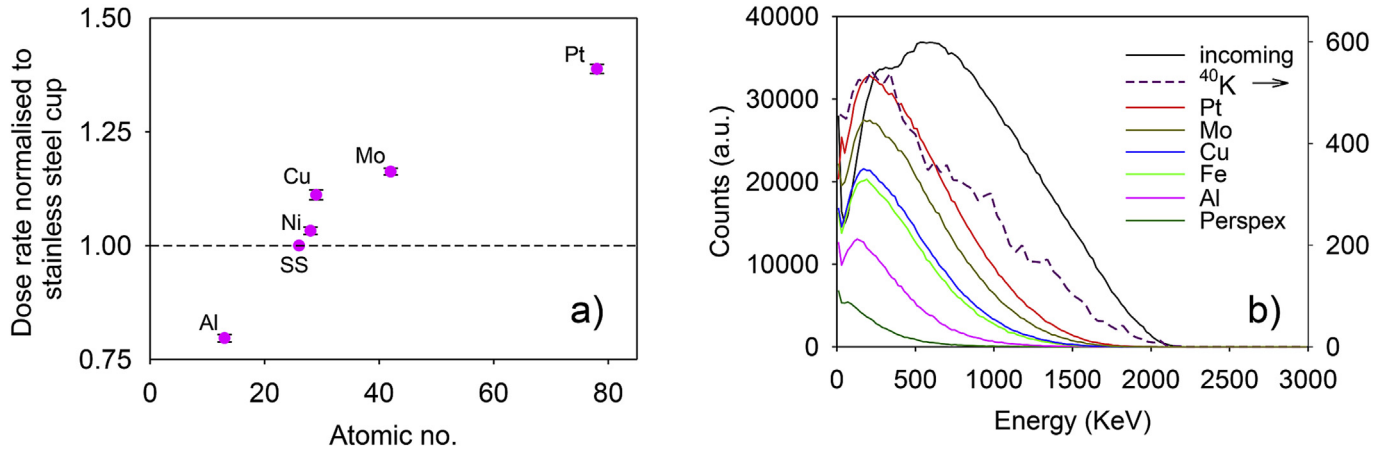

Fig. 3. a) β dose rate to quartz in cups as a function of atomic number normalised to that in conventional stainless-steel cups. (SS is derived using Fe) b) Modelled β source incident spectrum, and backscattered spectra from various infinitely thick substrates. Later modelling will use finite cup thickness and geometry, but this is expected to primarily reduce the intensities of the spectra.

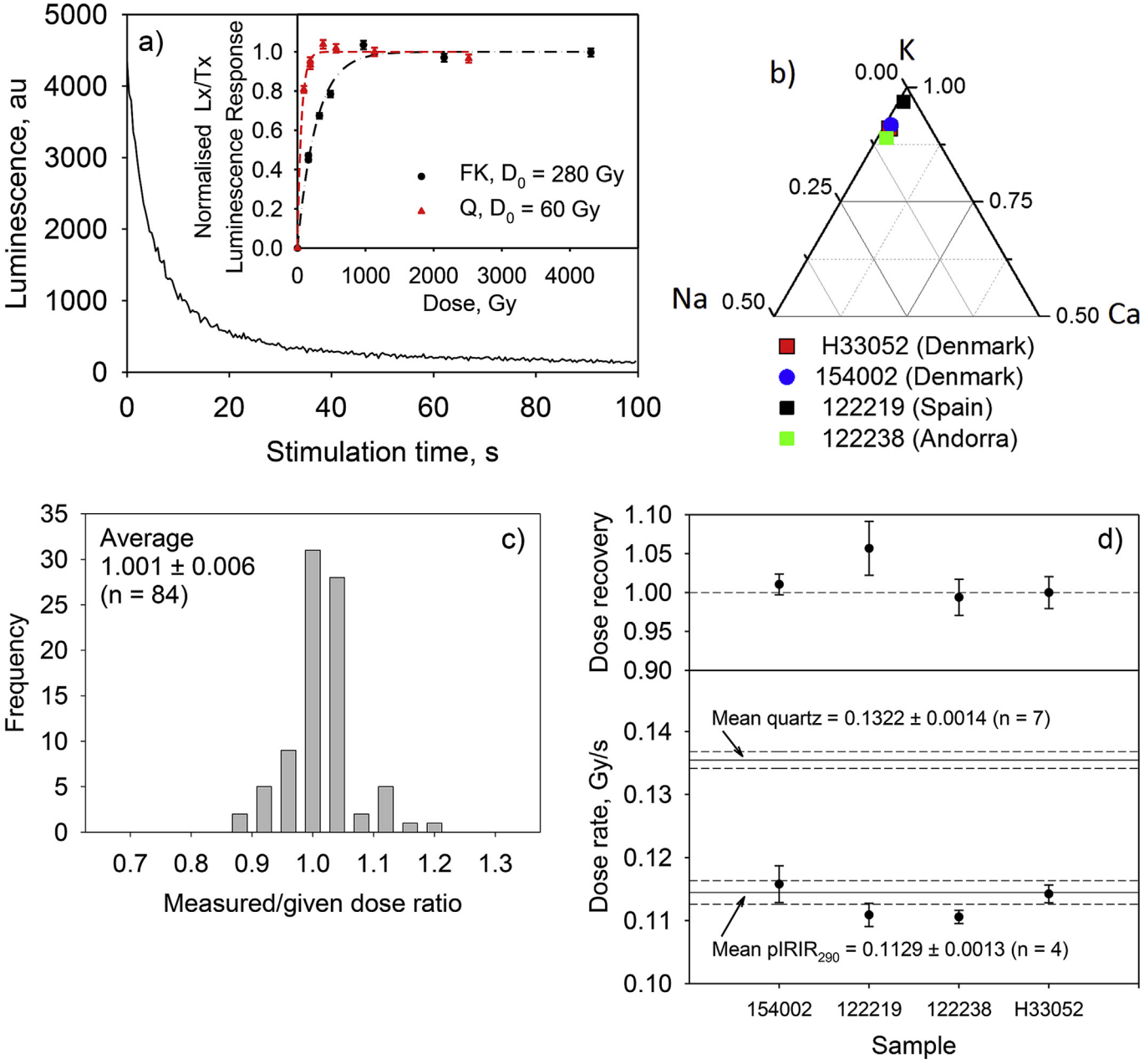

Fig. 4. a) A $pIRIR_{50,290}$ decay curve from a 2 mm large aliquot of K-feldspar from H33052 given a γ dose of 4.81 ± 0.07 Gy. The inset shows the normalised $pIRIR_{50,290}$ (black) and quartz OSL (red) dose response curves. b) Ternary diagram showing results of XRF-analyses of different K-feldspar extracts. c) Beta dose recovery histogram for K-feldspar from H33052 (annealed 550 °C/1 h). d) Dose recovery and apparent beta source dose rate for different K-feldspar extracts. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

5. K-feldspar

To test whether the observed over-dispersion in the quartz calibration time-series (Fig. S1) is related only to the luminescence characteristics of quartz or to variability in measurement procedures/instrumentation, we have begun to investigate the potential of using feldspar pIRIR signals for calibration.

From simple examination of mass absorption coefficients (NISTIR 5632) and stopping powers (NISTIR 4999), we anticipate that the beta dose rate to quartz and feldspar should be indistinguishable. To confirm this, we have used GEANT4 to formally model both the ratio of gamma dose rates in pure K feldspar and pure quartz (0.993 ± 0.002 (n = 5), averaged over a 1 mm thick slice) and the corresponding β dose rate ratio (1.0032 ± 0.0008, for a 200 μm thick slice of dosimeter placed on 0.5 mm stainless steel substrate). These data confirm that the β dose rate to feldspar should be within 1% of that to quartz.

K-feldspar was extracted ($\rho < 2.58\ g\ cm^{-3}$) from the same sediment H33052 (Rømø, Denmark) as is used for calibration quartz batch 90 onwards; a typical $pIRIR_{50,290}$ luminescence curve is shown in Fig. 4a (measured during IR stimulation with the sample held at 290 °C, after first preheating to 320 °C for 60s and then IR stimulation with the sample held at 50 °C); for reference, the normalised SAR dose response

curve is shown in the inset, compared to that of the calibration quartz derived from the same sample.

Before this K-rich feldspar extract can be used for calibration it must be shown to be pure, and we must test whether a laboratory β dose can be recovered using our chosen $pIRIR_{50,290}$ protocol. Fig. 4b shows the results of XRF analysis; the fraction of K-feldspar is > 90% for all samples. Fig. 4c shows the results of the β dose recovery test on sample H33052 after first heating to 550 °C for 1 h; the dose recovery ratio is indistinguishable from unity and the data have a relative standard deviation of ~5%.

A portion of this heated extract was then exposed to the ^{137}Cs gamma source as described above, and the β source calibrated in the usual manner, but using the $pIRIR_{50,290}$ signal. Rather disturbingly, the average apparent β source dose rate to feldspar is significantly lower (~15%) than that measured using standard calibration quartz (Fig. 4d, sample H33052).

As a further test of this unexpected discrepancy, three additional pure K-rich feldspar extracts from different locations (Fig. 4b) were prepared as above, and irradiated together in the ^{137}Cs γ source. Note that these three K-feldspar extracts also yield dose recovery ratios consistent with unity (Fig. 4d, top). The weighted mean of all four measurements is 0.1119 ± 0.0007 Gy/s (6 aliquots per sample). The data are not over-dispersed, but this is not surprising since all 24 aliquots were measured in a single sequence; the quartz over-dispersion arises mainly between sequences. Nevertheless, these dose rates are consistent with an average ratio of 0.854 ± 0.013 to that to quartz (Fig. 4d, bottom). The implications of this difference are considerable, and this unexpected discrepancy clearly requires further investigation. However, we can confidently rule out instability of the $pIRIR_{50,290}$ signal as a significant contribution, because this would tend to lead to an over-estimate of the β source dose rate, rather than an under-estimate.

6. Discussion and conclusions

It is clear that both the grain-size dependence and the backscatter contribution have the potential to contribute to multi-grain aliquot-to-aliquot over-dispersion during β irradiation. Both grain-size dependent attenuation effects on the backscattered and primary spectra, and the possibility of one grain sitting on top of another (and so experiencing a different backscatter contribution from grains sitting directly on the substrate) could lead to unexpected variability. The slope of the grain size dependency in Fig. 2b is $\sim 7 \times 10^{-4}\,\mu m^{-1}$ (for grain sizes > 100 μm); the grain-size range used here is 180–250 μm, suggesting an upper limit to the range in dose rates of ((250-180) x 7×10^{-4}) = 5%, about half of that observed. In practice, no samples will be entirely 180 μm or 250 μm, and so the real range in dose rates arising from grain size variation will be much smaller than this. The variation in dose rate arising from substrate variation is readily eliminated by ensuring a monolayer of grains on the substrate. Thus, we anticipate that, in practice for the multi-grain aliquots used in Fig. S1, the contributions to dose rate variability from grain size and substrate variations are likely to be negligible.

However, investigations into the effect of substrate variation do lead us to a conclusion of some practical importance. From modelling, we deduced that the net electron spectrum is likely to be closer to that experienced in nature for high Z substrates (although whether that is important is unknown). Thus it is concluded that there are unlikely to be any significant undesirable effects of using high Z substrates such as platinum (Pt) cups in routine irradiations, although it would be very important to ensure grains were presented as a monolayer. In contrast, there is likely to be significant practical benefit in using Pt cups; the average dose rate would be 40% greater than on stainless steel, and 75% greater than on Al.

The original motivation for investigating the β dose rate to feldspar was to test whether the poor reproducibility in quartz calibration arises because of instrument variability. At this stage in our investigations, we do not draw any conclusions on this. However, an unexpected difference of 15% in dose rate to quartz and feldspar has been observed. This is surprising because modelling suggests there should be no difference between feldspar and quartz calibrations. It is deduced that this difference must arise from differences in quartz and feldspar luminescence response to β and γ radiation (i.e. in χ_γ and χ_β for quartz and feldspar in Equation (1)). Given the observed reproducibility from sample to sample (in both Q and KF, Fig. 1 and Fig. 4d) this difference is likely to be due mainly to crystal structure.In addition, some grain-to-grain variation in dose rate within quartz and feldspar cannot be ruled out because of variations in both attenuation and backscatter.

Nevertheless, if this observation proves reliable, and if it applies to the natural β dose rate, then the effect on ages will be relatively small (< 5%) because the total beta dose rate component makes up about > 70% of the total. But this remains to be tested experimentally.

In summary.

1. The average apparent quartz dose rate is independent of the source of the quartz.
2. The effect of likely grain size and substrate variations on β dose rate reproducibility is probably not significant.
3. It may be possible to increase effective β source dose rates by ~40% using platinum instead of stainless steel cups.
4. The apparent β dose rate to feldspar is ~15% less than that to quartz, independent of origin; whether this translates into a 15% error in age depends on whether the same effect occurs in nature.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.radmeas.2018.05.014.

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